Docket No. 24180-124-004   **PATENT**

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In Re U.S. Patent Application of:	)
	) Art Unit: 3627
Applicant: CURIE *et al.*	)
	) Examiner: R. Dye
Serial No.: 09/901,427	)
	)
Filed: July 9, 2001	)
	)
For: TRANSPARENT MULTILAYER	)
POLYPROPYLENE CONTAINER	)
WITH BARRIER PROTECTION	)
	)

37 C.F.R. § 1.132
DECLARATION OF DR. ROBERT KNOLL

Commissioner of Patents and Trademarks
Washington, D.C. 20231

Dear Sir:

I, the undersigned Robert Knoll, Ph.D., declares the following:

1. I am an employee of Pechiney Plastics Packaging, Inc. and was a co-worker of the named inventors on the filing date of the present invention.

2. I have received a Bachelor of Science degree in Nuclear Engineering (1973), a Masters of Science degree in Nuclear Engineering (1974), another Masters of Science degree in Materials Science (1978), and a Ph.D. in Engineering Physics/Nuclear Engineering (1981), all from the University of Wisconsin at Madison on the dates respectively indicated.

3. I have over twenty years of experience as an innovative engineer and applied materials scientist. My experience as a materials scientist includes substantial research and analysis of polymers and polymer processing. For example, in my approximately eight years as a senior technical staff member at Johnson Controls, Inc., I conducted research and development

projects related to injection and biaxial large-strain deformation of polyethylene terephthalate (PET) and other polymers. In the approximately six years I have currently spent as a senior research associate of materials development with Pechiney Plastic Packaging, Inc., I have conducted research and development projects related to development, analysis and implementation of polymers into production lines to improve product performance and manufacturing efficiency, including blow molding PET and polypropylene (PP).

4. I have reviewed the August 13, 2003, Office Action in the above-identified patent application, as well as U.S. Patent No. 5,804,016 to Schmidt ("Schmidt") upon which that Office Action relies for its rejection of the pending claims.

5. I paid particular attention to paragraph 7 of the Office Action. I understand paragraph 7 to reject claims 25-28 and 30-41 because they are taught by Schmidt. I disagree. Specifically, paragraph 7 states as follow:

> Schmidt teaches multilayer containers made from polypropylene (col. 10, line 38) and having oxygen scavenging internal layers (col.12, lines 51-53). The outer, or "finish" layer, may be polyethylene terephthalate (col. 8, line 32). The containers are made by stretch blow molding (col. 7, lines 17-36). The performs from which the containers are made have support flanges (col. 9, line 25) and sidewalls that are 13 to 14.5 times thicker than the container made from them (col. 5, line 38). The preform has a flange on the bottom of the neck portion a thickened base and its bottom end is thinner than the upper base-forming portion of the preform (col. 5, lines 3-18). The average panel axial stretch is 3.0 to 3.2 (col. 5, lines 43-46). The preform may have an internal oxygen scavenging layer (col. 12, lines 51-53).

6. I consider myself one of ordinary skill in the art of injection stretch blowmolding of PET and PP.

7. I disagree that Schmidt teaches any polypropylene container to a person of ordinary skill in the art, as asserted in paragraph 7 of the Office Action.

8. I understand Schmidt to be directed to producing multilayer plastic containers having enhanced strength for high temperature and pressure applications. Schmidt indicates that his container comprises inner and outer layers of PET having with high intrinsic viscosity (IV), to withstand high temperature applications, and comprises the remainder of his container with a thick core layer of less expensive post-consumer PET ("PC-PET"). (Col. 3, lines 9-12) Schmidt also indicates that PC-PET typically has an IV lower than that of the high IV inner and outer layers and that layer separation occurs when the layers have an IV difference over a certain magnitude. (Col. 3, lines 13-22). As one of ordinary skill in the art, I understand that Schmidt considered layer separation to be an important commercial issue for carbonated soft drink containers (Col. 3, lines 23-25). I understand Schmidt to focus on how to construct a high temperature and high pressure multilayer container that will not suffer from layer separation.

As one of ordinary skill in the art, Schmidt teaches how to construct a high temperature and high pressure multilayer PET container that will not suffer from layer separation. Specifically, Schmidt teaches a preform injection method to overcome the layer separation problems caused by having layers having significantly different IV. At column 3, lines 30-36, Schmidt explains:

> It has been found that the injection molding and/or blow molding process conditions can substantially diminish or completely eliminate the problem of layer separation for IV deltas on the order of 0.10 or more. More specifically, the rate of injection and amount of pressure applied in the preform mold are increased to insure higher levels of layer bonding.

Schmidt continues at column 3, lines 54-59, by explaining the technical reason that his preform injection method overcomes the problems caused by the differences in IV:

> It is hypothesized that increasing the IV delta between the virgin PET and PC-PET alters the melt solubility of the materials sufficiently to reduce molecular migration and chain entanglement at the layer boundary, thus decreasing layer adhesion. The enhanced injection rate and pressure overcomes this problem.

Declaration of Dr. Robert Knoll

The Detailed Description Of The Invention confirms that Schmidt's focus is on using this injection method to overcome the layer separation problems caused by comprising layers of materials with different IVs.

9. Schmidt's claims reinforce my understanding that his invention is focused on this injection method. Every one of Schmidt's claims is directed to his injection method.

10. For these reasons, one of ordinary skill in the art would understand Schmidt to teach an injection method for preventing delamination in multilayer containers having layers with different IVs in order to facilitate cheap high temperature, high pressure containers.

11. I have reviewed Schmidt's disclosure of possible alternative materials at column 10, lines 35-54, which states as follows:

> For example, one or more layers of the preform and container, or portions thereof, can be made of various other polymers, such as polyolefins (e.g., polypropylene and polyethylene), polyvinyl chloride, polyarcylate, etc. Suitable polyesters include homopolymers, copolymers or blends of polyethylene terephthalate (PET), polybutylene terephthalate (PBT), polypropylene terephthalate (PPT), polyethylene napthalate (PEN), and a cyclohexane dimethanol/PET copolymer, known as PETG (available from Eastman Chemical Company, Kingsport, Tenn.). Polyesters based on terephthalic or isophthalic acid are commercially available and convenient. The hydroxy compounds are typically ethylene glycol and 1,4-di-(hydroxy methyl)-cyclohexane. In general, the phthalate polyester may include polymer linkages, side chains, and end groups not related to the formal precursors of a simple phthalate polyester previously specified. Conveniently, at least 90 mole percent will be terephthalic acid and at least 90 mole percent an aliphatic glycol or glycols, especially ethylene glycol.

12. As one of ordinary skill in the art, I understand this list of substitute materials to indicate that Schmidt's injection method may employ the listed materials, as substitutes for PET and PC-PET. It follows naturally that the listed substitutes are indicated for use with the injection method that is the focus of the invention. Schmidt does not, however, teach any of the modifications to the timing, temperature or pressure of his injection process. In fact, Schmidt

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teaches nothing about how to incorporate any of the listed alternative materials into his invention.

13. To be sure, Schmidt provides no directive or suggestion that his list of alternative materials are to be employed in the described preform that was designed to construct a PET container. One of ordinary skill in the art would not consider Schmidt to teach using PP, or any of the other materials it lists as possible alternatives, in a preform of an identical configuration to Schmidt's PET preform.

14. Rather, because Schmidt teaches an injection method, I understand Schmidt to teach that Schmidt's injection method may be used with any of the listed alternative materials. The injection method is the focus of the invention; the specific configuration of Schmidt's PET preform is not. I understand Schmidt's specific PET preform configuration to be a mere example of a working PET preform and incidental to his actual invention that is a preform injection method.

15. In fact, when Schmidt is read as a whole, it becomes apparent that Schmidt's invention is specific to PET. I understand that Schmidt's injection method identifies melt temperatures and material properties (e.g. IV) that are specific to PET. I understand that the "standard pressure" identified in Fig. 6 and described in column 6 is specific to PET, as is the "enhanced pressure" of Schmidt's invention. Likewise, I understand the preform configuration described by Schmidt to be specific to PET.

16. Having read both Schmidt and the Office Action, I understand the Office Action to rely on the features and characteristics of Schmidt's PET preform.

17. Intrinsic Viscosity ("IV") is a common measurement of PET and is typically identified by a vendor. A manufacturer uses the IV measurement to assist it in designing the processing

parameters for injection molding and blow molding. Neither I, nor others of ordinary skill in the art speak or think of PP in terms of IV. Moreover, to my knowledge the container industry does not employ IV measurements of PP in its design of PP containers or the processes by which they are constructed. Instead, PP is measured by its Melt Index, dictated by ASTM 1238.

18. Because necessity of Schmidt's injection method is premised on IV differential, I and others of ordinary skill in the art would not know how or when to apply the teachings of Schmidt to a container constructed from PP. I do not consider Schmidt's invention applicable to PP.

19. Given that persons of ordinary skill in the art speak of PP in terms of its Melt Index rather than it IV, one of ordinary skill in the art would not consider Schmidt applicable to PP, or know how to manufacture the high temperature, high pressure container of Schmidt from PP, despite the catch-all inclusion of PP in the laundry list of possible alternative materials.

20. Schmidt does not teach myself, or others of ordinary skill in the art, how to construct a high temperature and high pressure preform or container from PP or any of the other listed alternative materials. Schmidt provides no information about the dimensions, contours or stretch ratios necessary for any of the alternative materials. Neither do it provide an indication of the modifications necessary to use PP and achieve the objectives of the invention. Nowhere does Schmidt state that PP can be simply exchanged for PET without any other modifications to the details it provides for the preform and container.

21. If I, or others of ordinary skill in the art, were inclined to construct a PP container comporting with the teachings of Schmidt, I would not produce a PP preform having the detailed configuration of Schmidt's PET preform. Because of the different material properties of PET and PP, I would consider it necessary to modify the size, shape, thickness or other characteristic of the preform or container to accommodate the different material properties of PET and PP.

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22. For example PP has a tensile strength of between about 2990 and 5260 psi whereas PET has a tensile strength of about 6680 psi. PP has a modulus of elasticity of between about 156 and 185 Kpsi whereas PET has a modulus of elasticity of about 484 Kpsi. Those of ordinary skill in the art understand these properties to be very important considerations when designing a preform for injection and later blow molding into a container. A PET's IV measurement is also important as well as the corresponding Melt Index of a PP. I am not aware of a direct conversion between the IV of a PET and the Melt Index of a PP.

23. Thus, I and others of ordinary skill in the art would understand that the configuration of Schmidt's PET preform would require modification commensurate with the changes in material. Those of ordinary skill in the art would modify the configuration of the PET preform of Schmidt, if that person was inclined to change the material from PET to PP, in order to achieve the objective container of Schmidt.

24. All statements made herein of my own knowledge are true and all statements made on information and belief are believed to be true; and further these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both under Section 1001 of Title 18 of the United States Code, and such willful false statements may jeopardize the validity of any patent confirmed hereon.

Respectfully Submitted,

Date: _Nov. 6, 2003_   _Robert W. Knoll_

Robert Knoll, Ph.D.

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ASTM Designation: D 4603 – 91

Standard Test M th d f r Determining Inherent Viscosity of Poly(Ethylene Terephthalate) (PET)[1]

EXHIBIT

A

This standard is issued under the fixed designation D 4603; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method is for the determination of the inherent viscosity of poly(ethylene terephthalate) (PET) soluble at 0.50 % concentration in a 60/40 phenol/1,1,2,3-tetrachloroethane solution. Some highly crystalline forms of PET may not be soluble in this solvent mixture and will require a different procedure.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)[2]

D 446 Specification for Operating Instructions for Glass Capillary Kinematic Viscometers[2]

D 1972 Practice for Generic Marking of Plastic Products[3]

D 4019 Test Method for Moisture in Plastics by Coulometry[4]

E 1 Specification for ASTM Thermometers[5]

E 380 Practice for Use of the International System of Units (SI)[6]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[6]

2.2 *NIST Standard:*

C 602 Testing of Glass Volumetric Apparatus[7]

3. Terminology

3.1 Units, symbols, and abbreviations used in this test method are those recommended in Practice E 380.

3.2 *Descriptions of Terms Specific to This Standard:*

3.2.1 *inherent viscosity* (also known as the logarithmic viscosity number)—defined by the equation given in 11.1.

3.2.2 *PET*—as outlined in Practice D 1972, the PET acronym may be used to avoid trademark infringement and to comply with various state or federal laws.

4. Summary of Test Method

4.1 The inherent viscosity is determined by measuring the flow time of a solution of known polymer concentration and the flow time of the pure solvent in a capillary viscometer at a fixed temperature. The inherent viscosity value is calculated from the flow time values.

5. Significance and Use

5.1 The inherent viscosity is relatable to the composition and molecular weight of a polyester resin. It must be controlled so that the processability and end properties of the resin remain in a desired range.

6. Apparatus

6.1 *Cannon Ubbelohde Type 1B Viscometer*, as described in Specifications and Operating Instructions D 446.

6.2 *Viscometer Holder.*

6.3 *Electric Timer*, readable to 0.1 s, as described in Test Method D 445.

6.4 *Constant Temperature Bath*, control label at 30°C (86°F) ± 0.01°C (0.02°F).

6.5 *Kinematic Viscosity Thermometer ASTM 118* (for use at 30°C), conforming to Specification E 1.

6.6 *Temperature Controllable Magnetic Stirring Hot Plate.*

6.7 *TFE-Fluorocarbon Plastic-Coated Stirring Bars and a Magnetic Bar Retriever.*

6.8 *Volumetric Flasks and Stoppers*, 50-mL capacity, conforming to the standards of accuracy in NIST Circular No. C 602.

6.9 *Analytical Balance*, with readout to 0.0001 g.

6.10 *Borosilicate Funnels.*

6.11 *Stainless Steel Filter Screening*, 325-mesh or finer.

6.12 *Aspirator.*

6.13 *Wiley Mill Grinder*, with 20-mesh stainless steel screen.

6.14 *Drying Oven*, for equipment.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chem-

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ical Society where such specifications are available.[8] Other grades may be used, providing it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.[9]

7.2 *Phenol/1,1,2,2-tetrachloroethane Solution*, (PET), 60/40 weight % mixture (0.5 % moisture maximum, protected in such a manner as to maintain this maximum level). The PET should be protected against the degradation which can occur during the heating stage in 10.3 through the addition of a hydrogen chloride scavenger to the solvent, such as 0.2 weight % n-octyl-bis-(isoethyl mercaptoacetate).[11]

7.3 *Reagent Grade Methylene Chloride and Acetone*, rinsing solvents.

7.4 *Chromic Acid*, cleaning solution.

8. Hazards

8.1 The solvent used in this procedure is a mixture of 60 weight % phenol and 40 weight % 1,1,2,2-tetrachloroethane. Both compounds and the mixture are toxic and require care in handling. Reference should be made to the material safety data sheets available from the suppliers of these compounds for dealing with the hazards they present. In addition to using a hood for adequate ventilation in handling these materials, protection against skin contact is essential.

8.2 The material safety data sheets for methylene chloride, acetone, and the chromic acid solution used for cleaning the testing equipment should also be obtained from their suppliers and consulted before use.

9. Conditioning

9.1 If the sample of PET contains 0.5 % or more of inert material such as titanium dioxide or glass fiber, determine the amount of inert material accurately by a procedure suitable for the type of inert material present.

9.2 If the sample is suspected of being wet (in excess of the moisture level derived from exposure to ambient humidity conditions), dry the sample in an oven for a suggested minimum of 2 h at 65°C (149°F) ± 5°C (9°F) or until a constant weight of ± 0.1 % is reached. Moisture picked up from ambient humidity will weigh 0.30 % and can be neglected in this procedure. (See Note 1.)

NOTE 1—Test Method D 4019 or another suitable technical method may be used to determine moisture content.

9.3 If the sample is difficult to dissolve, it may be desirable to grind it to a 20-mesh screen size in a Wiley Mill. Care must be taken not to overheat the sample during the grinding operation. A low temperature can be maintained by grinding in the presence of dry ice or liquid nitrogen. Grind a 15 to 20 g sample, representative of the entire lot being tested. Drying may be necessary after the dry ice grinding step.

10. Procedure

10.1 Accurately weigh between 0.2475 and 0.2525 g (accurate to ±0.0002 g) of sample into a clean, dry 50-mL volumetric flask. If the sample contains more than 0.5 % inert material, weigh the amount of sample necessary to give the specified amount of PET.

10.2 Place a TFE-fluorocarbon plastic-coated stirring bar into the flask and add approximately 25 mL of solvent. Prepare one flask without any sample present. Cap the flasks.

10.3 Place the flasks in steel beakers and place on a magnetic hot plate which has been preheated to 110°C (230°F) ± 10°C (18°F). Heat the flasks for 15 min while stirring. Remove flasks and inspect for undissolved PET. If a sample does not dissolve completely, extend the stirring time for up to 30 more min while inspecting the sample at 10-min intervals. If the sample fails to dissolve completely at this time, this procedure is not applicable.

10.4 When the samples have completely dissolved, remove the flasks from the hot plate and allow them to cool to approximately room temperature. Remove the stirring bars with a magnetic retriever and wash the bar with fresh solvent, letting the wash solvent fall back into the volumetric flask. Add additional solvent to a level about 1 cm (0.4 in.) below the 50-mL mark. Place the flasks in the constant temperature bath preset at 30°C (86°F) ± 0.01°C (0.02°F). Allow the flasks to sit for 10 min to reach the bath temperature. Invert the stoppered flasks to wash down solvent droplets adhering to the flask walls above the polymer solution, and add sufficient solvent to raise the liquid level up to the 50-mL mark.

10.5 Pour the solution into a clean, dry, Cannon-Ubbelohde viscometer by passing it through a funnel and filter screen into the top of the larger viscometer tube. Fill the viscometer to a level between the level lines on the large reservoir bulb at the bottom of the larger tube. Remove the funnel and place the viscometer in the constant temperature bath preset at 30°C (86°F) ± 0.01°C (0.02°F). Allow at least 15 min for the temperature of the solution in the viscometer to reach equilibrium.

10.6 Using suction from an aspirator, draw the solution through the capillary to a level above the top calibration mark. Regulate the level by capping the breather tube with one rubber-gloved finger and carefully applying suction to the top of the capillary tube. Use care to prevent splashing or bubble formation. A valve in the aspirator line may be useful to control the suction.

10.7 Let the sample solution or solvent flow back down the capillary tube by removing the suction from the top of the capillary tube and by removing the finger from the top of the breather tube. The first flow is a rinse to wet the capillary bulb and finally equilibrate the sample solution to the bath temperature.

10.8 After the solution has drained out of the capillary, repeat 10.6 and 10.7 and time the period required for the liquid to fall back from the higher calibration mark to lower calibration mark above the capillary. Use the electric timer for this measurement. The bottom of the meniscus of the liquid surface is used for determining the times at which the liquid surface flows past the calibration marks.

10.9 Record the flow time and repeat the measurement three more times. Average these results unless the range in time exceeds 0.2 s, in which case make additional measurements until four within a range of 0.2 s are obtained for

[8] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analar Standards for Laboratory Chemicals", BDH, Ltd., Poole, Dorset, U.K., and the "United States Pharmacopeia."

[9] Available from M&T Chemicals, Inc., P. O. Box 1104, Rahway, NJ 07065.

averaging. Measure the solvent flow time in the same manner as the flow time of the solution samples.

10.10 During the measurements, record the bath temperature to the nearest 0.01°C (0.02°F). The range in temperature should not exceed 0.01°C.

10.11 When measurements are completed, remove the viscometer from the bath and carefully pour the solution from the viscometer into a safety disposal container.

11. Calculation

11.1 Determine the inherent viscosity as follows:

$$\eta_{inh} \frac{30°C}{0.5\%} = \frac{\ln \eta_r}{C}$$

where:

$\eta_{inh} \frac{30°C}{0.5\%}$ = inherent viscosity at 30°C (86°F) and at a polymer concentration of 0.5 g/dL (dimensions of inherent viscosity are dL/g),

η_r = relative viscosity = t/t_o,

t = average solution flow time, s,

t_o = average solvent flow time, s, and

C = polymer solution concentration, g/dL.

11.2 The intrinsic viscosity η can also be calculated from a single measurement of the relative viscosity using the Billmeyer relationship:[10]

$$\eta = 0.25 (\eta_r - 1 + 3 \ln \eta_r)/C$$

12. Report

12.1 Report the following information:

12.1.1 Sample identification and description,

12.1.2 Sample weight,

12.1.3 Percent of inert material,

12.1.4 Sample dissolution time and temperature,

12.1.5 Average solvent flow time,

12.1.6 Average solution flow time,

12.1.7 Average viscometer bath temperature,

12.1.8 Inherent viscosity (three significant places), and

12.1.9 Intrinsic viscosity (optional).

TABLE 1   Inherent Viscosity of PET at 30°C and 0.5 % Polymer Concentration

Material	Mean	S_r	S_R	I_r	I_R
2	0.6615	0.005755	0.01233	0.0163	0.0349
1	0.6949	0.007436	0.01369	0.0210	0.0387
3	0.7819	0.006819	0.01118	0.0193	0.0316

13. Precision and Bias[11]

13.1 Table 1 is based on a round-robin test conducted in 1984, involving three materials representing different inherent viscosity levels, tested by six laboratories. For each material, all the samples were prepared by one source. Each test result was based on two individual determinations. In each laboratory, two operators obtained one test result on each material each week for three weeks.

13.2 In Table 1, for the materials indicated, and for mean values that are derived from duplicate determinations:

13.2.1 S_r is the within-laboratory standard deviation of the mean, and I_r = 2.83 S_r. (See 13.2.3 for application of I_r.)

13.2.2 S_R is the between-laboratory standard deviation of the mean, and I_R = 2.83 S_R. (See 13.2.4 for application of I_R.)

13.2.3 *Repeatability*—In comparing two mean values for the same material, obtained by different operators using different equipment on different days in the same laboratory, the means should be judged not equivalent if they differ by more than the I_r value for that material and condition.

13.2.4. *Reproducibility*—In comparing two mean values for the same material, obtained by different operators using different equipment on different days in different laboratories, the means should be judged not equivalent if they differ by more than the I_R value for that material and condition.

13.2.5 Any judgment in accordance with 13.2.3 and 13.2.4 will have an approximate 95 % (0.95) probability of being correct.

NOTE 2—Other materials may give somewhat different results.

NOTE 3—For further information on the methodology used in this section, see Practice E 691.

13.3 *Bias*—Since no absolute test method exists for comparison, no bias statement can be presented for this test method.

14. Keywords

14.1 inherent viscosity; poly(ethylene terephthalate); PET

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[10] *Journal of Polymer Science*, Vol 4, 1949, p. 83.

[11] Supporting data are available from ASTM Headquarters. Request RR: D20-1132.

Designation: D 1238 – 90b

EXHIBIT

B

ALL-STATE LEGAL®

Standard Test Method for
Flow Rates of Thermoplastics by Extrusion Plastometer[1]

This standard is issued under the fixed designation D 1238; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers measurement of the rate of extrusion of molten resins through a die of a specified length and diameter under prescribed conditions of temperature, load, and piston position in the barrel as the timed measurement is being made.

1.2 Procedure A is a manual cutoff operation based on time used for materials having flow rates that fall generally between 0.15 and 50 g/10 min. Procedure B is an automatically timed flow rate measurement used for materials having flows from 0.50 to 900 g/10 min. By both procedures, the piston travel is generally the same during the timed measurement; the piston foot is about 46 and 20.6 mm (1.81 and 0.81 in.) above the die. Comparable flow rates have been obtained by these procedures in interlaboratory round-robin measurements of several materials described in 12.1.

NOTE 1—Round-robin testing indicates this test method may be suitable at flow rates up to 1500 g/10 min if the timing clock resolves the elapsed time to the nearest 0.01 s.

1.3 The values stated in SI units are to be regarded as the standard. The values in parentheses are given for information only.

1.4 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in Note 9.*

2. Referenced Documents

2.1 *ASTM Standards:*
D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing[2]
D 883 Terminology Relating to Plastics[2]
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]
2.2 *American National Standards Institute Standard:*
B46.1 on Surface Texture[4]

3. Terminology

3.1 *General:*

3.1.1 For definition of some of the technical terms used in this standard refer to Terminology D 883.

4. Significance and Use

4.1 This test method is particularly useful for quality control tests on thermoplastics.

NOTE 2—Polymers having flow rates less than 0.15 or greater than 900 g/10 min may be tested by the procedures in this test method; however, precision data have not been developed.

4.2 This test method serves to indicate the uniformity of the flow rate of the polymer as made by an individual process and, in this case, may be indicative of uniformity of other properties. However, uniformity of flow rate among various polymers as made by various processes does not, in the absence of other tests, indicate uniformity of other properties.

4.3 The flow rate obtained with the extrusion plastometer is not a fundamental polymer property. It is an empirically defined parameter critically influenced by the physical properties and molecular structure of the polymer and the conditions of measurement. The rheological characteristics of polymer melts depend on a number of variables. Since the values of these variables occurring in this test may differ substantially from those in large-scale processes, test results may not correlate directly with processing behavior.

4.4 The flow rate of a material may be measured under any of the conditions listed for it in 8.2. Additional characterization of a material can be obtained if more than one condition is used. In case two conditions are employed, a Flow Rate Ratio (FRR) may be obtained by dividing the flow rate at one condition by the flow rate at the other condition.

5. Apparatus

5.1 *Plastometer:*

5.1.1 The apparatus shall be a dead-weight piston plastometer consisting of a thermostatically controlled heated steel cylinder with a die at the lower end and a weighted piston operating within the cylinder. The essential features of the plastometer, illustrated in Figs. 1 and 2, are described in 5.2 to 5.8.

5.1.2 Relatively minor changes in the design and arrangement of the component parts have been shown to cause differences in results between laboratories. It is important, therefore, for the best interlaboratory agreement that the design adhere closely to the description herein; otherwise, it should be determined that modifications do not influence the results.

5.2 *Cylinder*—The steel cylinder shall be 50.8 mm (2 in.) in diameter, 162 mm (6⅜ in.) in length with a smooth, straight hole 9.5504 ± 0.0076 mm (0.3760 ± 0.0003 in.) in diameter, displaced 4.8 mm (³⁄₁₆ in.) from the cylinder axis.
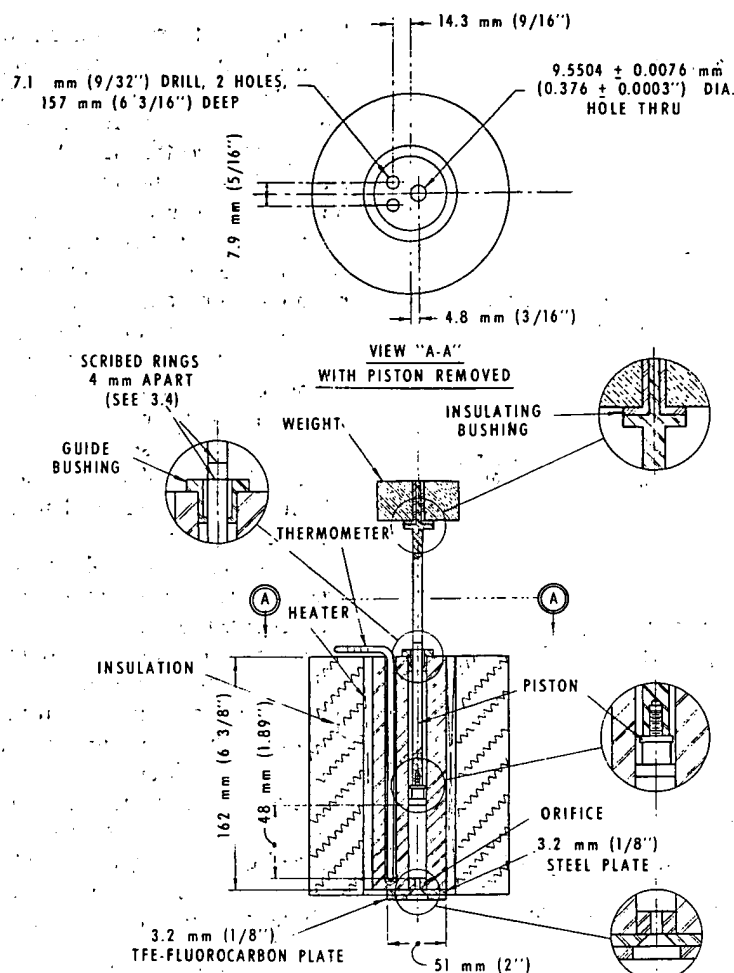
FIG. 1 General Arrangement of Extrusion Plastometer

Wells for a thermal sensor (thermoregulator, thermistor, etc.) and thermometer shall be provided as shown in Fig. 1. A 3.2-mm (⅛-in.) plate shall be attached to the bottom of the cylinder to retain the die. A hole in this plate, centered under the die and countersunk from below, allows free passage of the extrudate. The cylinder may be supported by at least two 6.4-mm (¼-in.) high-strength screws at the top (radially positioned at right angles to the applied load) or by at least two 10-mm (⅜-in.) diameter rods screwed into the side of the cylinder for attaching to a vertical support. The essential dimensions of a satisfactory cylinder of this type are shown in Fig. 1 (Note 3). The cylinder bore should be finished by techniques known to produce approximately 12 rms or better in accordance with ANSI B46.1.

Note 3—Cylinders made of SAE 52100 steel heat-hardened to a Rockwell hardness, C scale, of 60 to 64 give good service when used at temperatures below 200°C.[5] Cylinder liners of cobalt-chromium-tungsten alloy are also satisfactory to 300°C.

5.3 Die—The outside of the steel die shall be such diameter that it will fall freely to the bottom of the 9.5504-mm (0.3760-in.) diameter hole in the cylinder (Note 4). The die shall have a smooth straight bore 2.0955 ±

0.0051 mm (0.0825 ± 0.0002 in.) in diameter and shall be 8.000 ± 0.025 mm (0.315 ± 0.001 in.) in length. The bore and its finish are critical. It shall have no visible drill or other tool marks and no detectable eccentricity. The die bore shall be finished by techniques known to produce approximately 12 rms or better in accordance with ANSI B46.1.

Note 4—Die materials also found to be satisfactory are tungsten carbide,[6] synthetic sapphire, and cobalt-chromium-tungsten alloy.[5]

5.4 Piston:

5.4.1 The piston shall be made of steel with an insulating bushing at the top as a barrier to heat transfer from the piston to the weight. The land of the piston shall be 9.4742 ± 0.0076 mm (0.3730 ± 0.0003 in.) in diameter and 6.35 ± 0.13 mm (0.250 ± 0.005 in.) in length. The piston design may incorporate means for land replacement, for example, having threads and flats immediately above the land. Above the land, the piston shall be relieved to 8.890 ± 0.025 mm (0.350 ± 0.001 in.) in diameter (Note 5). The finish of the piston foot shall be 12 rms in accordance with ANSI B46.1.

Note 5—To improve standardization it is preferable that the piston be sleeved at the top and be relieved as described in 5.4.2.

Note 6—Pistons of SAE 52100 steel with the bottom 25 mm (1 in.),

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[5] Haynes Stellite 6B, manufactured by the Haynes Stellite Co., Kokomo, IN, has been found satisfactory for this purpose.

[6] Talide C-99, manufactured by the Metal Carbides Corp., Youngstown, OH, has been found satisfactory for this purpose.

9.5504 ± 0.0076 mm
(0.3760 ± 0.0003")

9.4742 ± 0.0076 mm
(0.3730 ± 0.0003")

8.890 ± 0.025 mm
(0.350 ± 0.001")

6.35 ± 0.13 mm
(0.250 ± 0.005")

8.000 ± 0.025 mm
(0.315 ± 0.001")

PISTON

CYLINDER
BORE

ORIFICE

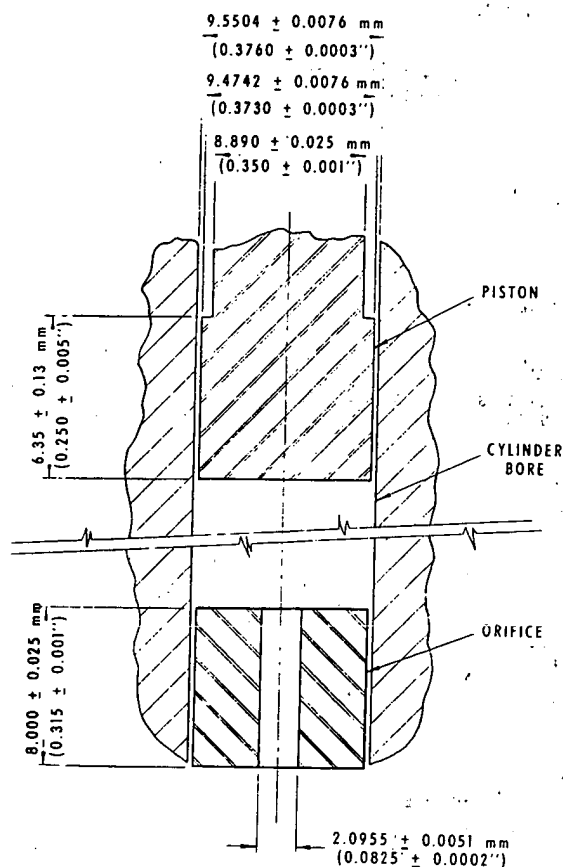2.0955 ± 0.0051 mm
(0.0825 ± 0.0002")

FIG. 2   Details of Extrusion Plastometer

including the foot, hardened to a Rockwell hardness. C scale, of 55 to 59 have been found to give good service when used at temperatures below 200°C.

5.4.2 A solid piston relieved to 7.900 ± 0.025 mm (0.311 ± 0.001 in.) in diameter above the land with a loose-fitting metal guiding sleeve at the top of the cylinder is desirable for use with the 21.6 kg load. If wear or corrosion is a problem, the piston should be of stainless steel and equipped with a detachable foot for ease of replacement.

5.4.3 For Procedure A the piston shall be scribed with two reference marks 4 mm apart in such fashion that when the lower mark coincides with the top of the cylinder or other suitable reference point, the bottom of the piston is 48 mm (1.89 in.) above the top of the die (see Fig. 1).

5.4.4 The combined weight of piston and load shall be within a tolerance of ±0.5 % of the selected load.

5.5 *Heater*:

5.5.1 Provision shall be made for heating the apparatus so that the temperature of the material can be maintained within ±0.2°C of the desired temperature during the test. At temperatures higher than 200°C, this degree of temperature control may be more difficult to obtain. The temperature specified shall be the equilibrium temperature of the material 12.7 mm (½ in.) above the die.

5.5.2 Calibrate the temperature-indicating device by means of a light-gage probe-type thermocouple or a platinum-resistance temperature sensor having a short sensing

length.[7] The thermocouple should be encased in a metallic sheath having a diameter of approximately 1.6 mm (¹⁄₁₆ in.) with its hot junction grounded to the end of the sheath. Insert the temperature sensor into the melt from the top of the cylinder so that it is 12.7 mm (½ in.) above the upper face of the die. The temperature sensors shall be used with a potentiometer having a sensitivity of at least 0.005 mV, or a temperature readout having a sensitivity of at least 0.1°C. It is desirable to calibrate the thermocouple or platinum-resistance temperature sensor and readout equipment with reference to a platinum resistance temperature thermometer. Comparison of the temperature sensor and readout of a calibrated thermometer similar to that described in 4.7, using the thermometer well containing heat-transfer medium, has also been found useful.

5.5.3 Heat shall be supplied by an electric band heater which covers the entire length of the cylinder. The heater may be a single- or multi-element heater, depending upon manufacturer's control means. The heater plus control system must be capable of maintaining the set points within the required ±0.2°C. If the heater contains two 100-W concentric elements, as close to 90 % as feasible of the power required to maintain the cylinder at the specified temperature should be supplied continuously by the outer of the two heating elements. Six tenths of this amount of power shall be applied intermittently by the inner element as required to maintain the specified temperature (Note 7). The cylinder with heater shall be lagged with 38 mm (1.5 in.) of foamed-glass insulation.[8] A TFE-fluorocarbon plate 3.2 mm (⅛ in.) in thickness shall be attached to the bottom of the cylinder to minimize heat loss at this point.

NOTE 7—Experience has shown that the correct adjustment of the power to the heaters is important in order that all instruments possess identical thermal characteristics. A convenient method for adjustment is obtained by measuring the voltage required to maintain the cylinder at the specified temperature (±1°C) using only the constant heater. Then the voltage to the constant heater is readjusted to the voltage obtained from the calculation:

$$E_c = \sqrt{0.9e^2} = 0.95e$$

where:

E_c = voltage required to maintain approximately 90 % of the power required to keep the cylinder at the specified temperature, and
e = voltage required to maintain the power required to keep the cylinder at the specified temperature.

The intermittent heater voltage is then obtained by the calculation:

$$E_i = \sqrt{0.6e^2} = 0.78e$$

where:

E_i = voltage specified for the intermittent heater, and
e = same as above.

Measurement of the voltages is imperative because variable autotransformer settings do not necessarily represent voltages.

5.6 *Temperature Controller*—The type of controller and sensor must be capable of meeting the required control tolerance specified in 5.5.1.

[7] Round-robin data showing flow rate and corresponding temperature profile of the melt obtained using probe-type thermocouples and platinum resistance temperature sensors can be obtained from ASTM Headquarters. Request RR:D20-1094.

[8] "Foamglas" 5.4-cm (2⅛-in.) diameter IW pipe insulation as manufactured by the Pittsburgh Corning Corp., Pittsburgh, PA, is recommended for this purpose.

NOTE 8—Wood's metal,[9] as a heat transfer medium, may be used with the indicating or control elements if required for accuracy or stability by a particular control system. Care should be taken that the Wood's metal is suitable for the selected temperature. Elements that could sustain damage upon cooling with Wood's metal should be removed prior to the cool down. Silicon oil, as a heat transfer medium, should be avoided based upon possible contamination of the extrusion plastometer cylinder and its effect on flow rate.

5.7 *Thermometer*—Thermometers having a range of 4°C graduated in 0.2°C divisions may be used to indicate temperature. The temperature at this point may not necessarily be the temperature of the material 12.7 mm (½ in.) above the die. The thermometer may be used to monitor indirectly the temperature of the material 12.7 mm (½ in.) above the die and may be calibrated by reference to a thermocouple inserted in the material 12.7 mm (½ in.) above the die. See 5.5.2 for description of thermocouple and method for measuring temperature.

NOTE 9—**Warning**: Caution should be observed with the use of a mercury-filled thermometer. Mercury vaporization occurs if the thermometer is broken. Mercury thermometers are not to be used at or above the boiling point of mercury, which is 357°C.

5.8 *Level*—Provision shall be made for vertical alignment of the bore of the extrusion plastometer. This is necessary to minimize subtractive loads resulting from rubbing or friction between the piston tip and sidewall. Means of alignment are discussed in Appendix X1.

5.9 *Accessory Equipment*—Necessary accessories include equipment for charging samples to the cylinder, a funnel, a die plug, a tool for cutting off the extruded sample, a timer or stop watch, cleaning equipment, and a balance accurate to ±0.001 g.

NOTE 10—Satisfactory operation of the apparatus for polyethylenes can be ascertained by making measurements on NIST Standard Reference Materials (SRM) Nos. 1475 and 1476.[10] These SRM polyethylenes are, respectively, a linear polyethylene having a flow rate of 2.07 g/10 min at test condition 190/0.325 and a branched polyethylene having a flow rate of 1.19 g/10 min at test condition 190/2.16 (melt index).

6. Test Specimen

6.1 The test specimen may be in any form that can be introduced into the bore of the cylinder, for example, powder, granules, strips of film, or molded slugs. It may be desirable to preform or pelletize a powder.

7. Conditioning

7.1 Many thermoplastic materials do not require conditioning prior to testing. Materials which contain volatile components, are chemically reactive, or have other special characteristics most probably require appropriate conditioning procedures. Moisture not only affects reproducibility of flow rate measurement but, in some types of materials, degradation is accelerated by moisture at the high temperatures used in testing. If conditioning is necessary, see the applicable material specification and Practice D 618.

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[9] Wood's Metal or an aluminum powder may be used. Wood's metal is available from Belmont Smelting and Refining Works, Inc., 320 Belmont Ave., Brooklyn, NY 11207.

[10] These standard polyethylenes are available from the National Institute of Standards and Technology, Office of Standard Reference Materials, Washington, DC 20234.

8. Procedural Conditions

8.1 Standard conditions of test are given in Table 1. The previously used alphabetized system for designating test conditions is shown in Table 1 for reference only. Test conditions shall be shown as: Condition ___/___, where the temperature in degrees Celsius is shown first, followed by the weight in kilograms. For example: Condition 190/2.16 (formerly Condition E).

8.2 The following conditions have been found satisfactory for the material listed:

Material	Condition	
Acetals (copolymer and homopolymer)	190/2.16	190/1.05
Acrylics	230/1.2	230/3.8
Acrylonitrile-butadiene-styrene	200/5.0	230/3.8
Cellulose esters	190/0.325	190/2.16
	190/21.60	210/2.16
Nylon	275/0.325	235/1.0
	235/2.16	235/5.0
	275/5.0	
Polychlorotrifluorethylene	265/12.5	
Polyethylene	125/0.325	125/2.16
	190/0.325	190/2.16
	190/21.60	190/10
	310/12.5	
Polycarbonate	300/1.2	
Polypropylene	230/2.16	
Polystyrene	200/5.0	230/1.2
	230/3.8	190/5.0
Polyterephthalate	250/2.16	210/2.16
	285/2.16	
Poly(vinyl acetal)	150/2.16	
Poly(phenylene sulfide)	315/5.0	
Styrene acrylonitrile	220/10	230/10
	230/3.8	
Styrenic Thermoplastic Elastomer	190/2.16	200/5.0
Thermoplastic Elastomer-Ether-Ester	190/2.16	220/2.16
	230/2.16	240/2.16
		250/2.16

8.3 If more than one condition is used and the material is polyethylene, the determination of Flow Rate Ratio (FRR) has been found to be useful. FRR is a dimensionless number derived by dividing the flow rate at Condition 190/10 by the flow rate at Condition 190/2.16.

NOTE 11—When determining such a ratio of flow rates for a material at the same temperature under different loads, it has been found that precision is maximized when one operator uses one Procedure (A or B), the same plastometer, and the same die for both measurements (the die need not be removed from the plastometer between the two determinations).

NOTE 12—Moisture sensitivity can significantly influence flow rate results for some materials. Appropriate material specifications should be referenced for specific pre-drying or sample handling instructions.

9. Procedure A—Manual Operation

9.1 Select conditions of temperature and load from Table 1 in accordance with material specifications such that flow rates will fall between 0.15 to 50 g/10 min.

9.2 Ensure that the bore of the extrusion plastometer is properly aligned in the vertical direction. (See Appendix X1).

9.3 The apparatus shall be clean. The parts are more easily cleaned while they are hot. The temperature of the cylinder with piston and die in place shall have been at the test temperature for at least 15 min before a test is begun. When the equipment is used repetitiously, it should not be necessary to heat the piston and die for 15 min. See Note 7 for procedure for adjusting temperature. Take care that cleaning or previous use shall not have changed the dimensions. Make frequent checks to determine whether the die

TABLE 1 Standard Test Conditions, Temperature, and Load

Condition		Temperature, °C	Total Load Including Piston, kg	Approximate Pressure	
Obsolete-For-reference only	Standard Designation			kPa	psi
A	125/0.325	125	0.325	44.8	6.5
B	125/2.16	125	2.16	298.2	43.25
C	150/2.16	150	2.16	298.2	43.25
D	190/0.325	190	0.325	44.8	6.5
E	190/2.16	190	2.16	298.2	43.25
F	190/21.60	190	21.60	2982.2	432.5
G	200/5.0	200	5.0	689.5	100.0
H	230/1.2	230	1.2	165.4	24.0
I	230/3.8	230	3.8	524.0	76.0
J	265/12.5	265	12.5	1723.7	250.0
K	275/0.325	275	0.325	44.8	6.5
L	230/2.16	230	2.16	298.2	43.25
M	190/1.05	190	1.05	144.7	21.0
N	190/10.0	190	10.0	1379.0	200.0
O	300/1.2	300	1.2	165.4	24.0
P	190/5.0	190	5.0	689.5	100.0
Q	235/1.0	235	1.0	138.2	20.05
R	235/2.16	235	2.16	298.2	43.25
S	235/5.0	235	5.0	689.5	100.0
T	250/2.16	250	2.16	298.2	43.25
U	310/12.5	310	12.5	1723.7	250.0
V	210/2.16	210	2.16	298.2	43.25
W	285/2.16	285	2.16	298.2	43.25
X	315/5.0	315	5.0	689.5	100.0

diameter is within the tolerances given in 5.3.

9.4 Remove the piston and place it on an insulated surface. Charge the cylinder within 1 min with a weighed portion of the sample according to the expected flow rate, as given in Table 2. Place the weighted piston in position, and start timing for a 6 to 8-min preheat.

NOTE 12—Experience has shown that for the best reproducibility the piston should operate within the same part of the cylinder for each measurement. The piston is scribed so the starting point for each extrusion is roughly the same. Some excess of material over the minimum required for the actual flow measurement portion of the test is provided by the charging weights shown in Table 2. This is necessary to achieve a void-free extrudate and flow equilibrium before start of rate measurements. For the lower flow rate materials, it may be necessary to manually force some of the excess resin out of the cylinder to obtain proper scribe mark position within the specified preheat time.

NOTE 13—There may be cases where 6 to 8 min of preheat time may not be sufficient. Six minutes is a minimum preheat time. Longer preheat periods are permissible when they can be shown to be necessary. If longer preheat times are used, the report shall so indicate. Care may be necessary to ensure that the sample so tested is adequately protected against changes in polymer structure, usually by appropriate antioxidant addition.

NOTE 14—It is frequently helpful to take interim cuts of the extrudate at uniform time intervals during the specified extrusion time. Weights of these individual cuts give an indication of the presence of bubbles which may be masked due to their size or to opacity of the sample. This technique is particularly helpful in the case of highly pigmented materials. Forcing out some of the resin manually during the preheat period often eliminates bubbles in the test extrudate.

9.5 For flow rates of 0.15 to 10 g/10 min, if the weighted piston alone does not purge sufficient material during the preheat time to obtain piston positioning as required in 9.6, manually purge the estimated excess prior to 4 min. The amount of purge must be regulated so subsequent free travel of the weighted piston will position the scribe marks at the reference start position between the 6th and 8th min (9.6). Manual purging in this manner is permissible only when it is

known that it will not affect results. Otherwise, purge sooner, or use a lower charge weight.

9.6 For flow rates of greater than 10 to 50 g/10 min, the loss of weighed sample as shown in Table 2 could be unduly large by proceeding as in 9.4 without modification. In this case, during the first 6 min of preheat time, either a piston support or a die plug may be used.

9.6.1 A piston support should be of such length that the lower scribe mark of the supported piston will be 25 mm (1 in.) above the top of the cylinder or other reference point (5.4.3). The support may be a piece of wooden dowel rod or the like of appropriate length beneath the weight, to support the weight and thus the piston in the desired position. The

TABLE 2 Standard Test Conditions, Sample Weight,[A] and Testing Time—Procedure A[B]

Flow Range, g/10 min	Suggested Weight of Sample in Cylinder, g	Time Interval, min	Factor for Obtaining Flow Rate in g/10 min
0.15 to 1.0	2.5 to 3.0	6.00	1.67
>1.0 to 3.5	3.0 to 5.0	3.00	3.33
>3.5 to 10	5.0 to 8.0	1.00	10.00
>10 to 25	4.0 to 8.0	0.50	20.00
>25 to 50	4.0 to 8.0	0.25	40.00

[A] This is a suggested weight for materials with melt densities of about 0.7 g/cm³. Correspondingly, greater quantities are suggested for materials of greater melt densities. Density of the molten resin (without filler) may be obtained using the procedure described by Terry, B. W., and Yang, K., "A New Method for Determining Melt Density as a Function of Pressure and Temperature," SPE Journal, SPEJA, Vol 20, No. 6, June 1964, p. 540 or the procedure described by Zoller, Paul, "The Pressure-Volume-Temperature Properties of Polyolefins," Journal of Applied Polymer Science, Vol 23, 1979, p. 1051. It may also be obtained from the weight of an extruded known volume of resin at the desired temperature. For example, 25.4 mm (1 in.) of piston movement extrudes 1.804 cm³ of resin. An estimate of the density of the material can be calculated from the following equation:

Resin density at test temperature = $W/1.804$

where W = weight of extruded resin.

[B] See 9.11.

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weighed sample as shown in Table 3 or use a piston support (see 9.6.1).

10.2.8 If it is necessary to force out excess resin manually so as to adjust the position of the piston in the cylinder, do this before 4 min of preheat. For expected flow rates greater than 10 g/10 min remove the piston stop or remove the plug at 6 min. Small deviation from the expected flow rate is permissible.

10.2.9 Upon subsequent free travel, the timer must be actuated between the sixth and eighth minute of total preheat time, otherwise discard the charge and readjust the charge weight.

10.2.10 Observe the extrudate for voids during the flow rate measurement time. Also note condition of the extrudate leaving the orifice for any indication of material leakage around the sides of the die.

10.2.11 Record the time to the nearest 0.1 s for the piston to complete the calibrated distance of travel. Discard any runs with voids occurring in the timed portion or leakage of extrudate around the die.

10.2.12 Discharge any remaining resin and clean die and cylinder as detailed in 8.8.

11. Calculation (Procedure B)

11.1 Calculate the flow rate in grams per 10 min as follows (Note 16):

$$\text{Flow rate} = (427 \times L \times d)/t$$

where:
L = length of calibrated piston travel, cm,
d = density of resin at test temperature, g/cm^3 (see reference under Table 2),
t = time of piston travel for length L, s, and
427 = mean of areas of piston and cylinder × 600.

NOTE 17—Factors that may be substituted in the following equation are given for some materials in Table 4.

$$\text{Flow rate, g/10 min} = F/t$$

where:
F = factor from Table 4, and
t = time of piston travel for length L, s.

NOTE 18—It has been found, however, that agreement between Procedures A and B may be optimized if an average melt density for a particular type material is determined with the actual equipment used and that value substituted into the equation given in 11.1.

12. Report

12.1 Report the following information:

12.1.1 Statement indicating the nature and physical form of the material charged to the cylinder.

12.1.2 Temperature and load at which the test is run shall be reported. The results and test conditions can be referred to as FR-condition, where the standard designation for the condition from Table 1 is shown (for example: FR-190/2.16).

NOTE 19—It has become customary to refer to the flow rate of polyethylene as "melt index" when obtained under Condition 190/2.16. However, for all other materials the use of melt index or any term other than "flow rate" is discouraged, regardless of the condition used.

12.1.3 Flow rate reported as the rate of extrusion in grams per 10 min.

12.1.4 Procedure used (A or B).

12.1.5 Any unusual behavior of the test specimen such as

TABLE 4 Factors for Calculation of Flow Rate

Material (Unpigmented)	Temperature, °C	Piston Travel, L, cm (in.)	Factor for Calculation of Flow Rate, F[A]
Polyethylene	190	2.54 (1)	828
Polyethylene	190	0.635 (0.25)	207
Polypropylene	230	2.54 (1)	801
Polypropylene	230	0.635 (0.25)	200

[A] Factors calculated using melt-density values of 0.7636 g/cm^3 for polyethylene and 0.7386 g/cm^3 for polypropylene, as expressed in article by Zoller, Paul, "The Pressure-Volume-Temperature Properties of Polyolefins," *Journal of Applied Polymer Science*, Vol 23, 1979, p. 1051. The base densities at 23°C for which the melt densities are reported were 0.917 g/cm^3 and 0.906 g/cm^3 for annealed low-density polyethylene and polypropylene homopolymer.

discoloration, sticking, extrudate surface irregularity or roughness, etc.

12.1.6 Details of conditioning, if any.

13. Precision and Bias (Procedures A and B)

13.1 *Precision:*

13.1.1 Tables 5 and 6 are based on a round robin[11] conducted in 1986 and 1987, involving polypropylene, polyethylene, polystyrene, polycarbonate and acrylic materials. The number of participating laboratories is shown for each material. Each laboratory tested two specimens for each material on three different days. The analysis in Practice E 691 is based on a test result being the average of two specimens.

13.1.2 Table 7 is based on a round robin[12] conducted in 1980 using Procedure B. Four polypropylene samples having flow rates from 250 to 1500 were tested in 9 laboratories.

NOTE 20: Caution—The following explanations of I_r and I_R (13.1.3 through 13.1.5) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Tables 5 to 7 should not be vigorously applied to acceptance or rejection of material since those data are specific to the round robin and may not be representative of other lots, conditions, materials or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials. The principles of 13.1.3 through 13.1.6 would then be valid for such data.

13.1.3 *Concept of I_r and I_R*—Relevant if S_r and S_R have been calculated from a large enough body of data, and if test results are averages obtained from testing two specimens.

13.1.4 *Repeatability, I_r*—In comparing two test results for the same material, obtained by the same operator using the same equipment on the same day, the two test results should be judged not equivalent if they differ by more than the I_r value for that material.

13.1.5 *Reproducibility, I_R*—In comparing two test results for the same material, obtained by different operators using different equipment on different days, the two test results should be judged not equivalent if they differ by more than the I_R value for that material.

13.1.6 Any judgment in accordance with 13.1.3 and 13.1.5 would have an approximate 95 % (0.95) probability of being correct.

13.2 *Bias*—There are no recognized standards by which to estimate bias of this test method.

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[11] Supporting data are available from ASTM Headquarters. Request RR: D-20-1164.

[12] Supporting data are available from ASTM Headquarters. Request RR: D-20-1124.

TABLE 5 Precision, Procedure A (values in g/10 min)

Material	Condition	Average	S_r^A	S_R^B	I_r^C	I_R^D	No. of Laboratories
Polyethylene	190/2.16	0.27	0.008	0.022	0.023	0.063	9
Polyethylene	190/2.16	0.40	0.012	0.038	0.035	0.108	9
Polyethylene	190/2.16	2.04	0.026	0.079	0.073	0.224	9
Polyethylene	190/2.16	44.1	0.919	1.232	2.560	3.486	7
Polypropylene	230/2.16	2.23	0.106	0.226	0.299	0.639	9
Polypropylene	230/2.16	7.09	0.222	0.471	0.627	1.331	9
Polypropylene	230/2.16	32.8	0.581	1.051	1.644	2.974	9
Polystyrene	200/5	1.67	0.024	0.122	0.068	0.344	6
Polystyrene	200/5	8.82	0.190	0.667	0.538	1.886	6
Polystyrene	200/5	13.3	0.305	0.925	0.864	2.617	6
Polycarbonate	300/1.2	2.41	0.076	0.115	0.215	0.326	4
Polycarbonate	300/1.2	10.5	0.429	0.647	1.215	1.830	4
Polycarbonate	300/1.2	16.2	0.155	1.109	0.438	3.140	4
Acrylic	230/3.8	2.59	0.051	0.051	0.145	0.145	3

[A] S_r = within-laboratory standard deviation of the average,
[B] S_R = between-laboratories standard deviation of the average,
[C] I_r = 2.83 S_r, and
[D] I_R = 2.83 S_R.

TABLE 6 Precision, Procedure B (values in g/10 min)

Material	Condition	Average	S_r^A	S_R^B	I_r^C	I_R^D	No. of Laboratories
Polyethylene	190/2.16	0.27	0.009	0.014	0.026	0.039	8
Polyethylene	190/2.16	0.40	0.016	0.027	0.045	0.076	8
Polyethylene	190/2.16	2.04	0.040	0.094	0.112	0.266	9
Polyethylene	190/2.16	43.7	0.997	1.924	2.819	5.443	8
Polypropylene	230/2.16	2.25	0.052	0.214	0.1466	0.604	8
Polypropylene	230/2.16	7.16	0.143	0.589	0.4051	1.666	8
Polypropylene	230/2.16	32.6	0.693	0.945	1.959	2.672	8
Polystyrene	200/5	1.65	0.037	0.166	0.106	0.470	4
Polystyrene	200/5	8.39	0.144	0.423	0.406	1.197	4
Polystyrene	200/5	13.0	0.108	0.387	0.306	1.097	4

[A] S_r = within-laboratory standard deviation of the average,
[B] S_R = between-laboratories standard deviation of the average,
[C] I_r = 2.83 S_r, and
[D] I_R = 2.83 S_R.

TABLE 7 Precision, Procedure B (values in g/10 min)

Material	Condition	Average	S_r^A	S_R^B	I_r^C	I_R^D
Polypropylene	230/2.16	245	13.2	16.6	37.4	46.9
Polypropylene	230/2.16	482	31.8	40.0	89.9	113
Polypropylene	230/2.16	837	20.9	58.6	59.1	166
Polypropylene	230/2.16	1603	129	243	365	688

[A] S_r = within-laboratory standard deviation of the average,
[B] S_R = between-laboratories standard deviation of the average,
[C] I_r = 2.83 S_r, and
[D] I_R = 2.83 S_R.

APPENDIX

(Nonmandatory Information)

X1. EXTRUSION PLASTOMETER BORE ALIGNMENT

X1.1 A fixture consisting of a circular level mounted on a shaft having two bearing points 9.47 + 0.00 − 0.0076 mm (0.373 + 0.00 − 0.0003 in.) in diameter that can be inserted into the bore has been found suitable. A circular level that can be rigidly mounted on the piston rod for insertion into the bore may also be satisfactory. A circular level having a sensitivity of 20 minutes per 2.5 mm (0.1 in.) has been found satisfactory. Other alignment techniques that give comparable alignment sensitivity would be considered satisfactory.

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